

SPATIAL DISTRIBUTION AND POTENTIAL TOXICITY ASSESSMENT OF SELECTED PAHs IN BOTTOM SEDIMENTS OF POLISH RESERVOIRS

Agnieszka JANCEWICZ, Urszula DMITRUK & Urszula TOMCZUK

Institute of Meteorology and Water Management – National Research Institute, Dam Monitoring Centre, Podleśna 61, 01-673 Warsaw, Poland, e-mail: agnieszka.jancewicz@imgw.pl

Abstract: Water reservoirs are a natural area of sedimentation of pollutants (e.g. PAHs, PCBs or heavy metals) transported by the river and carried from the drainage basin. Polycyclic aromatic hydrocarbons (PAHs) in samples of bottom sediments gathered from six reservoirs located on the territory of Poland were analysed. The adverse effects of the sediments to aquatic organisms from PAHs were determined by using Sediment Quality Guidelines (SQG). Based on the studies, it was found that the PAHs contents in the analysed sediments were varied, and the total values of 7 PAHs (Σ_7 PAHs) range from 24 to 12103 $\mu\text{g kg}^{-1}$ dry weight (d.w.). The highest concentration were observed in reservoirs Złotniki and Bukówka located in the south-western industrial area of Poland. The lowest total PAH concentrations were found in sediments of Nielisz (57-155 $\mu\text{g kg}^{-1}$ d.w.) and Klimkówka (54-220 $\mu\text{g kg}^{-1}$ d.w.) from rural and forest areas. A comparison of the PAH concentrations with the ERL (effect range – low) and ERM (effect range – medium) indicates that in more than 50% of samples adverse biological effect may occasionally occur. But only in 2 samples PAHs concentrations were greater than ERM. In a few sample of sediments from Tresna and Rybnik reservoirs individual PAH concentrations above ERL value were observed.

Key words: PAHs, Sediment, Reservoir, Sediment Quality Guidelines, Toxicity assessment, Poland

1. INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) are a large group of persistent organic compounds (Li W.H. et al., 2008). They are a natural component of crude oil and its by-products, they are produced in volcanic eruptions, forest fire and in the process of biosynthesis. Their widespread presence in the environment is largely the result of human activity (van Drooge et al., 2011). PAHs are formed and emitted as a result of the incomplete combustion of organic energy sources (Akyüz & Çabuk, 2008; Chen et al., 2012; Khalili et al., 1995). The main source of the formation of PAHs is the petrochemical, coal and steel, energy industries as well as coal consumption for home heating, automobile traffic and waste incinerators.

PAHs are discharged to the aquatic environment mainly with atmospheric deposition, emitted gases and industrial wastewaters (Guerra, 2012; Patrolecco et al., 2010). Other sources of these contamination are the surface run offs from roads, cities, rural areas, emergency spills of crude oil and its by-products and also polluted sewage sludge (Achten & Hofmann,

2009; Dmitruk et al., 2012, Ünlü et al., 2010; Zawieja & Wolski, 2013). Due to the poor solubility in water and high affinity for solid particles, a substantial portion of PAHs is adsorbed on the suspension and is accumulated in bottom sediments (Li B. et al., 2012; Srogi, 2007; Vinãs et al., 2010).

Due to disturbance to the hydrological regime of rivers, dam reservoirs are areas of increased accumulation of suspension and related contaminants including PAHs. Sedimentation is a complex process which has its source in catchment area of the reservoir. The debris material carried by the river is the result of the side and bottom erosion and drainage area denudation (Kosik et al., 2012). The accumulation of sediments leads to a permanent reduction of their capacity. Polish water reservoirs lost $168 \cdot 10^6$ m³ of their initial capacity during the mean time of operation 45 years (Kosik et al., 2012). Inorganic substances constitute more than 90% of the accumulated material and the rest are organic substances whose concentration increases from a backflow region in the direction of a dam (Wiejaczka, 2011).

In order to evaluate sediment contamination,

many methods for determining the quality standards of bottom sediments have been developed worldwide. Those methods enable to assess the impact of sediments on living organisms, depending on concentrations of hazardous compounds contained therein (Burton, 2002; Long et al., 1995; MacDonalds et al., 2000; Swartz, 1999). Effect range – low (ERL) and effect range – medium (ERM) are commonly used methods for assessing freshwater sediments (Hahladakis et al., 2013; He et al., 2014; Li W.-H. et al., 2012; Malik et al., 2011; Panagiotopoulos et al., 2010). The ERL and ERM values are intended to define chemical concentration ranges that are rarely (<ERL), occasionally (between ERL and ERM), or frequently (>ERM) associated with adverse biological effects.

2. STUDY AREA

The work involved an analysis of sediments collected from six reservoirs located in southern Poland (Fig. 1). The study reservoirs are supplied with water from basins of different sizes and different physiographic and economic nature. These are multi-purpose objects, the functions of which include: flood protection, water supply, water power engineering and recreation. Based on the location and nature of the rivers on which they are built, they can be divided into three groups: Sudeten, Carpathian and lowland reservoirs. Detailed information about the study reservoirs is shown in table 1.

Sudeten reservoirs are located on the rivers in the Oder River basin, in the most industrialized region in Poland. Sudeten rivers are short watercourses characterized by large drops and rapid water level rise. This group includes the Bukówka (B) reservoir located at 268+500 km of the Bóbr River and the Złotniki (Z) reservoir located at 91+700 km of the Kwisa River. Carpathian reservoirs are located in the mountainous regions of rivers in the Vistula River basin. The rivers are characterized by large drops, substantial flows and fluctuations in water levels as well as intensive bank and riverbed erosion processes. The study group of Carpathian reservoirs included: the Klimkówka (K)

reservoir located at 54+400 km of the Ropa River and the Tresna (T) reservoir (known also as Lake Żywiec) located at 40+020 km of the Soła River. And the last group, lowland reservoirs are located on rivers with low longitudinal slopes and broad, meandering riverbeds. In its major proportion, debris material is composed of sand of varying granulation. Lowland reservoirs include: Nielisz (N) (235+200 km of the Wieprz River) and Rybnik (R) (22+800 km of the Ruda River). The Rybnik reservoir consists of the main reservoir and expansion reservoirs, separated by several side dams along the reservoir banks.

3. MATERIALS AND METHODS

3.1. Sampling of sediments

In 2010, 54 samples of bottom sediments were collected from 6 reservoirs and the concentrations of the following PAHs: benzo(a)anthracene (BaA), benzo(b)fluoranthene (BbF), benzo(k)fluoranthene (BkF), benzo(a)pyrene (BaP), indeno(1,2,3-c,d)pyrene (IP), dibenzo(a,h)anthracene (DA) and benzo(g,h,i)perylene (BP) were determined. The location of reservoirs and the sampling sites is shown in figure 1.

Collected samples of sediments weighing 1 kg each have been placed in closed glass containers and transported to the laboratory at a temperature of 4°C. Firstly the sediment samples were dried in darkness at room temperature and sieved with a 2 mm nylon sieve. Next, fraction with a particle size of < 2 mm was pounded in an agate mortar to obtain a crushed sample (grains of < 0.063 mm).

3.2. PAHs analysis

20 g of sediments mixed with 20 g of anhydrous sodium sulphate (POCH, Poland) were extracted for 8 hours in a Soxhlet apparatus by 200 ml of n-hexane (Merck, Germany). This method of extraction is one of the major techniques for PAHs determination in sediment samples (Yamaguchi & Lee, 2010).

Table 1. Detailed information about the study reservoirs.

Reservoir	River	Year of start of operation	Capacity at max. impoundment	Area at max. impoundment	Drainage area
			•10 ⁶ m ³	ha	km ²
Bukówka	Bóbr	1988	16.8	199	58
Złotniki	Kwisa	1924	71.8	130	288
Klimkówka	Ropa	1995	43.5	306	210
Tresna	Soła	1967	11.2	964	1030
Nielisz	Wieprz	1976/2007	25.6	742	1236
Rybnik	Ruda	1972	25.8	545	308

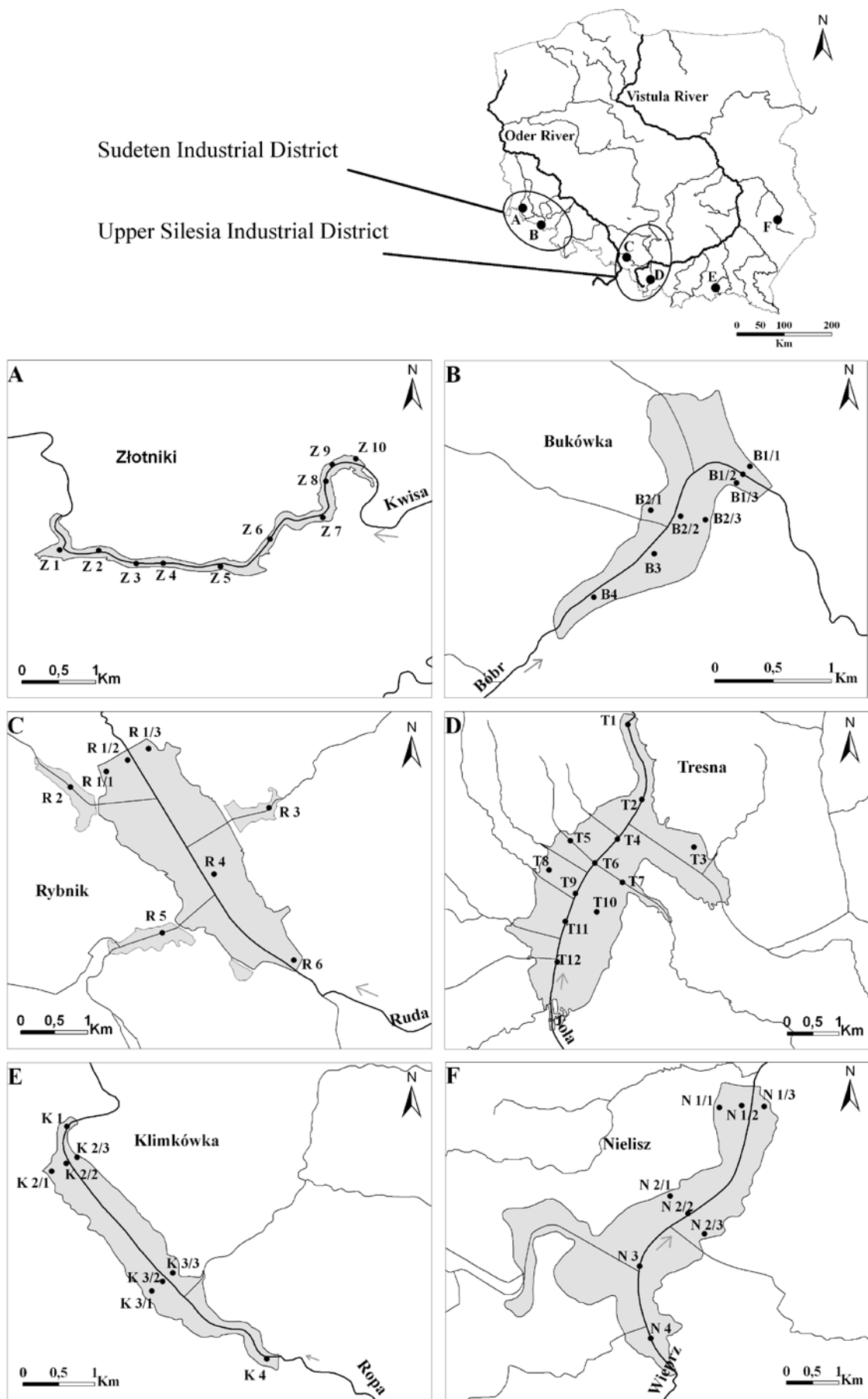


Figure 1. Location of the sediment sampling sites.

The extract was concentrated to about 2 ml in a rotary evaporator and then the extract was fractionated with a silica gel column (Merck, Germany). A mixture of dichloromethane (Merck, Germany) and n-hexane (1/1, v/v) was used for elution of PAHs. The eluted fractions were concentrated to dryness under a gentle stream of nitrogen at 40 °C. The dry remains were dissolved in 1 ml of n-hexane and the solution was analyzed.

The determination of PAHs was performed on an Agilent 6890 gas chromatography 5973 mass selective detector (GC-MS, Agilent, USA) system. A HP-5MS capillary column (30 m x 0.25 mm x 0.25 µm) was used. The oven temperature increased from an initial temperature of 50 °C to 200 °C by 20 °C min⁻¹ and held for 1 minute, heated 10 °C min⁻¹ to 250 °C, held 7 minutes and finally heated 10 °C min⁻¹ to 300 °C and held for 2 minute. Calibration of the GC-MS system was performed for all 7 PAHs. Supelco PAH reference material (10 µg ml⁻¹) was used as the calibration solution. The regression coefficient changed from 0.997 to 0.999. The average recoveries of PAHs ranged from 87% to 109%.

4. RESULTS AND DISCUSSION

4.1. Sediments quality parameters

The sediments quality parameters (pH, organic matter OM, total nitrogen TN and total phosphorus TP) were determined in all 54 samples. Concentrations of these parameters of the different reservoirs vary among them, as depicted in table 2. Concentrations of OM were in the range of 8.7 – 331 g kg⁻¹ d.w. (dry weight) with the lowest value at Rybnik and the highest at Nielisz. Maximum and minimum mean values were also found in this two reservoirs and were 2.7% and 14 % respectively. In other reservoirs the mean OM concentrations ranged from 6 to 8 %, except Złotniki where was 12%. The concentrations of OM were high correlated to TN with correlation coefficient 0.9, whereas had no relationship with TP. Nutrients mainly come from industrial wastewater, domestic sewage, agricultural non-point source pollution and surface run-off. Content of TN ranged from 100 mg kg⁻¹ d.w. to 1655 mg kg⁻¹ d.w. The lowest concentration was found in expansion reservoir of Rybnik (sampling point R5), while the highest was observed in Nielisz (N2/3). The relatively high levels of OM and TN in Nielisz sediments could be mainly attributed to rural activities in that area. TP values in sediments of five analysed reservoirs were higher than the TN values. The exception was Nielisz, where the mean values of TN and TP were 883 mg kg⁻¹ d.w. and 527 mg kg⁻¹ d.w. respectively. Highest concentrations of TP were found

in Złotniki reservoir and ranged from 371 to 2663 mg kg⁻¹ d.w. with mean value of 1286 mg kg⁻¹ d.w.

Table 2. The sediments quality parameters

		pH	OM	TN	TP
		-	g kg ⁻¹	mg kg ⁻¹	mg kg ⁻¹
B	Mean	6.4	78	469	751
	Median	6.4	81	462	718
	Range	5.9-7.0	34-105	252-651	396-1345
Z	Mean	6.3	122	673	1286
	Median	6.4	147	696	1099
	Range	5.6-6.6	14-170	126-1113	371-2663
K	Mean	6.7	62	369	467
	Median	6.6	59	337	392
	Range	5.9-7.4	48-90	318-598	305-853
T	Mean	7.3	80	409	850
	Median	7.3	88	450	770
	Range	6.9-7.7	24-93	120-506	512-1378
R	Mean	7.1	27	268	406
	Median	7.1	21	241	310
	Range	6.8-7.6	8.7-55	100-658	78-976
N	Mean	7,2	142	883	527
	Median	7,3	106	724	468
	Range	7.1-7.4	47-331	432-1655	361-890

4.2. PAH concentrations

The contents of PAH varied widely among the six reservoirs. The total concentrations of PAH (Σ_7 PAHs) were found in range of 24 µg kg⁻¹ d.w. to 12103 µg kg⁻¹ d.w. with the mean 1579 µg kg⁻¹ d.w. The lowest value of Σ_7 PAHs was determined on the right site of Rybnik reservoir, near the dam. The highest concentrations were determined in the Sudeten reservoirs – Bukówka and Złotniki, where mean concentrations of Σ_7 PAHs were 3118 µg kg⁻¹ d.w. and 4621 µg kg⁻¹ d.w. respectively (Table 3). The highest concentration was found in the Bukówka reservoir; the peak level was recorded at the sampling site B1/1 - on the left side of the reservoir, near the dam (Fig. 1B). Sediments collected from the Bukówka reservoir were the most diverse in terms of the total concentration of PAHs and the standard deviation there was more than 3800 µg kg⁻¹ d.w. The similar content of PAHs (9062 µg kg⁻¹ d.w.) was found in Złotniki at sampling site Z7. The high PAH concentrations in these two reservoirs could be result of industrial land use in south-western Poland (Sudeten Industrial District). A unique feature of this region is a large concentration of small and large industrial facilities in both urban and rural areas (energy, electromechanical, building materials industries and textiles). The long-term anthropogenic pressure has led to significant environmental pollution in the mainly a coal-mining area of the Upper Silesian Industrial District where Rybnik and Tresna reservoirs are located.

Table 3. Mean, median, standard deviation (SD) and range of PAHs concentrations in study sediments.

$\mu\text{g kg}^{-1}$ d.w.		BaA	BbF	BkF	BaP	IP	DA	BP	$\Sigma_7\text{PAHs}$
B	Mean	623	466	596	677	301	69	385	3118
	Median	334	290	303	354	164	50	233	1716
	Range	69-2687	91-1578	81-2327	88-2848	68-1093	32-195	66-1375	497-12103
	SD	868	502	751	920	344	55	440	3870
Z	Mean	725	803	840	876	614	93	672	4621
	Median	746	848	888	931	551	92	672	4570
	Range	159-1358	123-1504	139-1627	157-1718	81-1346	28-162	95-1364	782-9062
	SD	426	482	520	543	446	45	436	2877
K	Mean	18	24	20	21	19	11	18	131
	Median	18	24	22	21	19	11	18	134
	Range	7-28	10-45	8-32	8-35	8-32	4-17	8-33	54-220
	SD	9	13	10	11	9	6	9	64
T	Mean	170	121	167	171	71	17	95	811
	Median	147	106	154	155	62	15	83	723
	Range	75-340	73-234	87-308	98-286	44-115	11-41	63-150	461-1319
	SD	75	46	67	60	23	8	33	294
R	Mean	50	52	53	52	40	25	39	310
	Median	20	27	25	21	20	12	21	146
	Range	3-176	4-161	4-180	4-177	3-122	3-66	3-114	24-997
	SD	60	55	60	61	43	26	41	345
N	Mean	13	18	17	18	16	7	15	104
	Median	13	17	17	18	15	7	14	103
	Range	7-24	9-27	9-27	9-27	8-25	6-8	8-20	57-155
	SD	5	6	7	7	6	1	5	36

There are also numerous power stations in the area. Emissions of air pollutants from individual household heating may also have a significant impact on the environmental pollution (Guerra, 2012; Patrolecco et al., 2010). PAH concentrations in the sediments of this two reservoirs were lower than in the Sudeten ones, and $\Sigma_7\text{PAHs}$ ranged from 24 to 997 $\mu\text{g kg}^{-1}$ d.w. in Rybnik and from 461 to 1319 $\mu\text{g kg}^{-1}$ d.w. in Tresna, with mean values of 310 and 811 $\mu\text{g kg}^{-1}$ d.w. respectively. The mean contents of individual PAHs analysed (with the exception of DA) in the sediments of the Tresna reservoir were about twice as high as those in the Rybnik reservoir (Table 3). As expected the lowest concentrations of PAHs were observed in the Klimkówka and Nielisz reservoirs where the mean values of $\Sigma_7\text{PAHs}$ amounted to 131 $\mu\text{g kg}^{-1}$ d.w. and 104 $\mu\text{g kg}^{-1}$ d.w. These reservoirs are located in non-urbanized areas. The Nielisz reservoir basin is dominated by areas used for agricultural purposes (Jancewicz et al., 2012), while the Klimkówka reservoir is located in forest areas (Wiejaczka, 2011) which affect the low concentrations of PAHs in the environment.

The maximum of the concentration of individual PAH were found in Złotniki sediments. The mean and median showed relatively similar values (Table 3) while the standard deviations showed high values indicating large variations between minimum and maximum contents of this compounds. In Bukówka and Rybnik reservoirs mean

values were twice as high as the medians with the standard deviation in range from 55 $\mu\text{g kg}^{-1}$ d.w. (DA) to 868 $\mu\text{g kg}^{-1}$ d.w. (BaA) in Bukówka and from 26 $\mu\text{g kg}^{-1}$ d.w. (DA) to 61 $\mu\text{g kg}^{-1}$ d.w. (BaP) in Rybnik. In Sudeten reservoirs BaP was the dominant PAH and its contents amounted to 2848 $\mu\text{g kg}^{-1}$ d.w. in Bukówka and to 1718 $\mu\text{g kg}^{-1}$ d.w. in Złotniki. In the other analysed sediments mean concentrations of individual PAHs were on the similar levels within reservoir, the exception was DA that occurred at contents approximately twice lower than other PAHs.

The PAHs distribution was different in analysed reservoirs. In bottom sediments of Bukówka, Klimkówka and Nielisz increasing trend of individual PAH values with the decreasing distance from the dam was observed. The highest concentrations of all seven PAHs were noted in sampling points near the dam: B1/1; K1; N1/2. Opposite situation was recorded in Tresna sediments. Maximum contents of BkF, BaP, IP and BP were found in the middle part of the reservoir (T9) and the rest tree PAHs in point T3. At sampling point located near the dam (T1) the lowest concentrations of almost all analysed PAHs were observed (exceptions: BkF – T12; DA – T2, T5). In case of Rybnik most contaminated sediments were found in expansion reservoir on the east side (R3). In the main reservoir the highest concentrations occurred at sampling point R4 and were at least 25% lower than at R3.

Table 4. Comparisons of PAH contents ($\mu\text{g kg}^{-1}$ d.w.) in different European reservoir and river sediments.

$\mu\text{g kg}^{-1}$ d.w.		BaA	BbF	BkF	BaP	IP	DA	BP	References
Velke Kozmalovce Reservoir (Slovakia)	Range	654-2440	336-678	173-382	294-636	194-282	26-44	119-193	Hiller et al., 2009
	Mean	1671	512	282	473	223	33	145	
Zemplinska Sirava Reservoir (Slovakia)	Range	1-27	8-70	2-27	5-55	6-71	3-9	12-48	
	Mean	14	46	19	35	44	5.8	31	
Arc River (France)	Range	9.6-244	7.3-142	2.3-43	0.7-105	2-82	0.6-45	1.2-78	Kanzari et al., 2012
	Mean	-	-	-	-	-	-	-	
Ebro River (Spain)	Range	-	4-994	1.7-1762	1.4-941	1.6-335	-	3-412	Navarro et al., 2006
	Mean	-	129	121	93	66	-	61	
Tiber River (Italy)	Range	-	23-52	12-24	12-32	51-100	-	11-24	Patrolecco et al., 2010
	Mean	-	-	-	-	-	-	-	
Bosna River (Bosnia & Herzegovina)	Range	14-3545	-	31-6257	1.4-3691	14-2275	2.4-546	12-1499	Harman et al., 2013
	Mean	-	-	-	-	-	-	-	
Vistula River (Poland)	Range	52-560	56-736	28-480	56-488	26-320	12-336	28-488	Dmitruk et al., 2008
	Mean	192	294	161	216	135	92	138	
Danube River (Hungary)	Range	<0.2-94	-	-	<0.2-58	<0.2-46	<0.2-1.4	<0.2-26	Nagy et al., 2013
	Mean	16	-	-	11	6.2	1.3	4	

The physical-chemical characterize of sediments (e.g. OM, grain size) often influence the transport and fate of PAHs (Li B. et al., 2012). The Pearson correlation analysis showed significant correlation between individual PAH concentrations and the sediment OM contents in Złotniki and Klimkówka (exception DA). The correlation coefficients in both reservoirs range between 0.76 – 0.87 ($p < 0.05$). In the other reservoirs OM contents was not important in the distribution of PAHs in sediments.

A lot of previous researches have been reported about PAHs levels in other sediments in rivers, lake and reservoirs of the world. A direct comparison of literature data is difficult due to difference in analytical method, the thickness of the collected sediment etc. However comparison of the PAHs concentrations observed for polish reservoirs with other of the world was attempted. Table 4 shows the level of PAH concentrations found in the bottom sediments of European reservoirs and rivers. Mean PAHs levels (except BaA) in Bukówka and Złotniki reservoirs were higher than those reported in Velke Kozmanowice sediments in Slovakia – highly industrialized catchment area (Hiller et al., 2009). Higher than in polish reservoirs maximum levels for BaA, BkF, BaP, DA and BP were detected by Harman et al., (2013) in Bosna River. Range of PAHs values in Klimkówka and Nielisz were comparable with those from Zemplinska Sirava reservoir (Hiller, 2009), Tiber River (Patrolecco et al., 2010) and Danube River (Nagy et al., 2013). The sedimentary PAHs contaminated levels in Tresna reservoir was

lower compared with findings in Vistula River (Dmitruk et al., 2008) but similar to those reported in Spain – Ebro River (Navarro et al., 2006).

5. APPLICATION OF SQGs

In order to provide an ecotoxicological aspect of sediment contamination, concentrations of individual PAH present in this study (BaA, BbF, BkF, BaP, DA) were compared with available sediment quality guidelines (SQG) for fresh water ecosystems. The ERL and ERM were developed from an extensive database of biological effects determined by a variety of methods (for example: toxicity tests, field observations, other sediment guidelines). The dry weight normalized database for each PAH compound was sorted and the ERL and ERM identified as the lower 10th (ERL) and 50th (ERM) percentiles of effects data (Swartz, 1999).

The results from application of SQG for selected PAH are shown in table 5. In case of all PAHs, none of the sampling points in non-urbanized areas (Klimkówka, Nielisz) exceeded the SQG values. In other four reservoirs concentrations of investigated PAHs presented in this study were higher than ERL value in many localisations. Samples taken from Bukówka and Złotniki were more contaminant with PAH than the ones taken from other reservoirs (Fig. 2). Concentrations ranging between the ERL and ERM were found in about 70% of sediment samples from the Złotniki and 50% of sediment samples from the Bukówka.

Table 5. Risk assessments of PAHs according to the sediment quality criteria, $\mu\text{g kg}^{-1}$ (Burton, 2002).

PAH	ERL	ERM	ERL-ERM	>ERM
BaA	260	1600	B2/1; B2/2; B2/3; B4; Z2-Z9; T3; T9	B1/1
BbF	320	1880	B1/1; B2/1; B2/2; B2/3; Z2-Z9	-
BkF	280	1620	B2/1; B2/2; B2/3; Z2-Z6; Z8; Z9; T9	B1/1; Z7
BaP	430	1600	B2/1; B2/3; Z2; Z3; Z5; Z6; Z8; Z9	B1/1; Z7
DA	63,4	260	B1/1; B2/1; Z2; Z3; Z5-Z9; R3	-

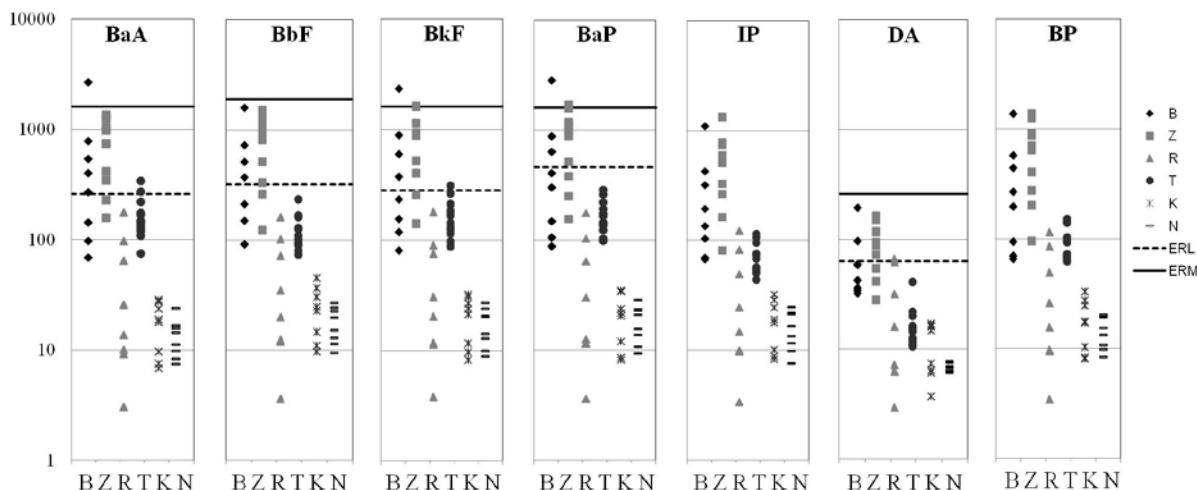


Figure 2. Concentrations of PAHs in all bottom sediment samples from six reservoirs (B – Bukówka, Z – Złotniki, R – Rybnik, T – Tresna, K – Klimkówka, N – Nielisz), in comparison with SQG (ERL and ERM), $\mu\text{g kg}^{-1}$.

In this two reservoirs values above which adverse effect would frequently occur ($> \text{ERM}$) were observed in sampling site B1/1 (BaA, BkF, BaP) and in Z7 (BkF, BaP). The noted concentrations of BaA and BkF in sediments of Tresna reservoir (T3; T9) were in range between ERL and ERM and it mean that toxicity effect may occasionally occur. In Rybnik reservoir only concentration of DA (sampling site R3) were slightly above the ERL.

6. CONCLUSIONS

In summary this study demonstrated contamination of bottom sediments present in reservoirs in different regions of Poland. The concentrations of PAHs varied greatly among six reservoirs and it can be assumed that their content largely depends on industrialization of the catchment area. The highest concentrations of PAHs were found in the Sudeten reservoirs: Bukówka and Złotniki located in highly industrialized area, and the lowest in Klimkówka (Carpathian reservoir) and Nielisz (lowland reservoir), located in forest and agricultural areas. Comparing the contents of PAH in the sediments of Polish reservoirs with the values presented in the literature, it has been found that they are on a similar level to concentrations observed in aquatic sediments from other parts of Europe. Assessment of sediment contamination using Sediment Quality Guidelines

(ERL, ERM) inferred that PAHs in sediments of investigated reservoirs might cause adverse biological effects rarely or occasionally.

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